इंटरनेट मानक

Disclosure to Promote the Right To Information

Whereas the Parliament of India has set out to provide a practical regime of right to information for citizens to secure access to information under the control of public authorities, in order to promote transparency and accountability in the working of every public authority, and whereas the attached publication of the Bureau of Indian Standards is of particular interest to the public, particularly disadvantaged communities and those engaged in the pursuit of education and knowledge, the attached public safety standard is made available to promote the timely dissemination of this information in an accurate manner to the public.

"जानने का अधिकार, जीने का अधिकार"
Mazdoor Kisan Shakti Sangathan
"The Right to Information, The Right to Live"

"पुराने को छोड नये के तरफ"
Jawaharlal Nehru
"Step Out From the Old to the New"

IS 13322 (1992): Furfural, Technical [PCD 9: Organic Chemicals Alcohols and Allied Products and Dye Intermediates]

"ज्ञान से एक नये भारत का निर्माण"
Satyanarayan Gangaram Pitroda
"Invent a New India Using Knowledge"

Act Number 22 of 2005
The Indian Parliament
RIGHT TO INFORMATION

Made Available By
Public.Resource.Org

"ज्ञान एक ऐसा खजाना है जो कभी चुराया नहीं जा सकता है"
Bhartṛhari—Nītiśatakam
"Knowledge is such a treasure which cannot be stolen"

BLANK PAGE

BRUTUM FULMEN

IGNORANTIA JURIS NON EXCUSAT

PROTECTED BY COPYRIGHT

IS 13322 : 1992

(Reaffirmed 2004)

भारतीय मानक

फर्फ्यूरल, तकनीकी — विशिष्टि

Indian Standard

FURFURAL, TECHNICAL — SPECIFICATION

UDC 547·724·1

© BIS 1992

BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

February 1992 **Price Group 4**

Organic Chemicals (Miscellaneous) Sectional Committee, PCD 9

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Organic Chemicals (Miscellaneous) Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

Furfural finds extensive use as a solvent in refineries to extract the aromatic components from lube base stock to improve the viscosity index of the lube oil. Large quantities of furfural are also used in the preparation of furane resins such as furfural phenyl resins, furfural acetone resins, furfuryl alcohol resins, etc. It is an important chemical intermediate used for the manufacture of wide variety of organic chemicals such as tetrahydrofuran, adipic acid, hexamethylene diamine, nylon, furfuryl alcohol, maleic anhydride, nitrofuranes pyrole, tetramethylene glycol etc,

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

FURFURAL, TECHNICAL — SPECIFICATION

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for furfural, technical.

2 REFERENCES

The following Indian Standards are necessary adjuncts to this standard:

IS No.	*Title*
1070 : 1977	Water for general laboratory use (*second revision*)
2303 : 1963	Method for grading glass for alkalinity
2620 : 1963	Distilling flasks
4905 : 1968	Methods for random sampling

3 REQUIREMENTS

3.1 The material shall be colourless liquid free from suspended matter. It shall be soluble in alcohol, ether, benzene and water.

3.2 The material shall also comply with the requirements prescribed in Table 1 when tested according to the methods given in Annex A. Reference to the relevant clauses of Annex A is given in col 4 of the Table 1.

4 PRECAUTIONS IN HANDLING

4.1 Furfural is handled in bulk or drums and its flammability is comparable to kerosene. Fire may be controlled by the use of water, foam, carbon dioxide or powder extinguishers. Furfural, being a powerful solvent and penetrant, the containers, tanks, joints and valves must be tight and in good condition. Gasketing material should be either 'TEFLON' or 'VITON'. If drums are being filled or emptied they must be earthed.

Furfural can be stored in steel tanks without any special precautions. There is a gradual darkening of colour and increase in acidity, water and polymer formation when furfural is stored for long periods in contact with air, auto oxidation can be prevented by storage in an oxygen free atmosphere.

4.2 Furfural has been used for many years with no adverse reports to the health of those handling it, but like all organic liquids it must be treated with caution. Good ventilation should be provided and skin contact avoided. If contacted with skin, it should be washed off promptly with soap and water and any clothing which is contaminated would be removed immediately. Any contact with the eyes must be treated immediately by washing with copious amount of water and

Table 1 Requirements for Furfural, Technical

(*Clauses* 3.2, B-4 *and* B-5)

Sl No.	Characteristic	Requirement	Method of Test (Ref to Cl No. in Annex A)
(1)	(2)	(3)	(4)
i)	Specific gravity, at 27°C/27°C	1·152 to 1·154	A-2
ii)	Acidity (as CH_3COOH), percent by mass, *Max*	0·20	A-3
iii)	Water, percent by mass, *Max*	0·20	A-4
iv)	Distillation range	The difference between the temperatures (running points) at which 2 and 98 percent of the volume taken have been collected shall not exceed 3°C. The range shall include 161·7°C.	A-5
v)	Ash, percent by mass, *Max*	0·005	A-6

then receiving medical attention as soon as possible.

5 PACKING AND MARKING

5.1 Packing

The material shall be packed in 200 litres steel drums under inert atmosphere using carbon dioxide or nitrogen.

5.2 Marking

The containers shall be marked legibly and indelibly with the following information:

a) Name of the material,

b) Net mass,

c) Indication of the source of manufacture,

d) Batch number, and

e) Date of manufacture.

5.2.1 The containers may also be marked with the Standard Mark.

6 SAMPLING

The method of drawing representative test samples of the material and the criteria for conformity shall be as prescribed in Annex B.

ANNEX A

(*Clause* 3.2 and *Table* 1)

METHOD OF TEST FOR FURFURAL, TECHNICAL

A-1 QUALITY OF REAGENTS

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070 : 1977) shall be used in tests.

NOTE — 'Pure chemicals' shall mean that chemicals that do not contain impurities which affect the results of analysis.

A-2 DETERMINATION OF SPECIFIC GRAVITY

A-2.1 Apparatus

A-2.1.1 *Pyknometer or Relative Density Bottle* — 25-ml capacity.

A-2.1.2 *Water-Bath* — Maintained at 27±0·2°C

A-2.2 Procedure

Clean and dry the Pyknometer or relative density bottle. Weigh and fill it with freshly boiled distilled water. Place it in the bath maintained at 27 ± 0·2°C, and allow sufficient time (about 45 minutes) to attain the temperature of the bath. Insert the capillary stopper which has also been brought to 27 ± 0·2°C. Wipe the excess liquid from the stopper, remove the Pyknometer or the relative density bottle from the bath, bring to room temperature and weigh. Empty the Pyknometer or the relative density bottle, clean and dry it, and repeat the operation with the material at 27 ± 0·2°C.

A-2.3 Calculation

$$\text{Specific gravity at } 27^\circ\text{C}/27^\circ\text{C} = \frac{M_1 - M_2}{M_3 - M_2}$$

where

M_1 = mass in g of the Pyknometer or relative density bottle filled with the material,

M_2 = mass in g of the dry Pyknometer or relative density bottle, and

M_3 = mass in g of the pyknometer or relative density bottle filled with water

A-3 DETERMINATION OF ACIDITY

A-3.0 Principle

Acidity is determined by titrating with standard sodium hydroxide solution, using phenolphthalein as indicator.

A-3.1 Reagents

A-3.1.1 *Standard Sodium Hydroxide Solution* — 0·1 N.

A-3.1.2 *Sulphuric Acid Solution* — Approximately 0·05 N.

A-3.1.3 *Phenolphthalein Indicator* — Dissolve 0·5 g of phenolphthalien in 100 ml of 95 percent (*v/v*) ethanol and make faintly pink by the addition of dilute sodium hydroxide solution.

A-3.2 Apparatus

A-3.2.1 *Burette* — capacity 10-ml, graduated in 0·02 ml divisions.

A-3.3 Procedure

Place approximately 200 ml of water in a 500-ml conical flask and add 0·5 ml of the phenolphthalein solution. Neutralize the water by addition of the standard sodium hydroxide solution or the sulphuric acid solution as required. Add 10·0 ml of sample, swirl the flask to dissolve and titrate using a burette with the standard sodium hydroxide solution, until the appearance of a pale pink colour which persists for at least 10 to 15 seconds.

A-3.4 Calculation

$$\text{Acidity (as } CH_3\,COOH\text{), percent by mass} = \frac{V_1 \times N \times 6}{V_2 \times \rho}$$

where

V_1 = volume, in millilitre, of standard sodium hydroxide solution used for the titration;

V_2 = volume in ml of sample taken;

ρ = density, in g/ml, of the sample at 27°C (*see* A-2); and

N = normality of sodium hydroxide solution.

A-4 DETERMINATION OF WATER CONTENT

A-4.1 Reagents

A-4.1.1 *Toluene* — anydrous.

A-4.2 Apparatus

The apparatus is essentially a ground glass flask of 500-ml capacity fitted with a reflux condenser and a device permitting to condense the vapours and measure the volume of condensed water in a graduated collector. A standard Dean and Stark assembly with a 2-ml receiver with 0·02 ml accuracy is suitable for the purpose.

A-4.3 Procedure

Clean and dry the apparatus. Place 100 ml each of furfural and toluene in the distillation flask, add small pieces of pumice and connect to the rest of the apparatus. Fill the calibrated receiver with toluene by pouring down the condenser. Turn on the cooling water and then heat the distillation flask. Adjust the distillation rate to about 2 drops per second initially increasing to 4 drops per second after most of the water has been distilled. Rinse down condenser with toluene and check to be sure that no more water distills over. (The total time of distillation should be about 60 minutes and the time of reflux should be about 45 minutes.) Stop distillation, rinse the condenser thoroughly with toluene and brush down any water droplets adhering to the walls by means of a small policeman attached to a glass rod. Allow the receiver to cool to room temperature and read the volume of water collected.

A-4.3.1 *Calculation*

$$\text{Moisture, percent by mass} = \frac{V}{\rho}$$

where

V = volume in ml of water collected, and

ρ = density in g/ml of furfural sample.

A-5 DETERMINATION OF DISTILLATION RANGE

A-5.1 Apparatus

A-5.1.1 *Distillation Flask* — *see* IS 2620 : 1963.

A-5.1.2 *Thermometer* — of partial immersion type, so fitted in the flask that the bottom of the capillary is in level with the lower edge of the side-tubes joint and the immersion mark is level with the top of the cork.

A-5.1.2.1 The recommended dimensions, tolerances and graduations of the thermometer are as follows:

Range	95° to 255°C
Graduation	0·5 deg
Longer lines at each	1 deg and 5 deg
Fully figured at each	100 deg
Fractional figuring at each	2 deg and 10 deg
Immersion	100 mm
Overall length, *Max*	335 mm
Length of main scale, *Min*	190 mm
Bulb length	15 to 20 mm
Stem diameter	5·5 to 8·0 mm
Distance from bottom of bulb to bottom of main scale, *Min*	125 mm
Distance from bottom of bulb to top of contraction chamber, *Max*	35 mm
Maximum error	1·0

NOTE — The thermometer shall bear a certificate from the National Physical Laboratory, New Delhi or by any other organization authorized by the Government of India to issue such a certificate.

A-5.1.3 *Liebig Condenser* — Made of Type 1 glass (graded according to IS 2303 : 1963) with a wall thickness of 1·0 to 1·5 mm conforming to the shape and dimensions given in Fig. 1.

A-5.1.4 *Receiver* — 100-ml capacity, with the dimensions and graduations shown in Fig. 2.

A-5.1.5 *Rectangular Draught Screen* — made of 0·8 mm thick metal sheet with the dimensions shown in Fig. 3 and open at top and bottom. It shall comply with the following requirements:

a) In each of the two narrow sides of the draught screen, there shall be two circular holes, each of 25 mm diameter, and in each of the four sides of the draught screen, there shall be three holes with their centres 25 mm above base of the draught screen. These holes shall occupy the position shown in Fig. 3, the diameter of each of the holes centrally situated in the wider sides shall be 25 mm, and of the remaining ten holes shall be 12·5 mm. At the middle of each of the wider sides, a vertical slot with the dimension shown in Fig. 3 shall be cut down from the top of the screen. A removal shutter conforming to the dimensions shown in Fig. 4 shall be provided for closing the vertial slot not in use.

b) A shelf of hard asbestos, 6 mm in thickness, having a central circular hole 110 mm in diameter, shall be supported horizontally in the screen to ensure that hot gases from the source of heat do not come in contact with the sides or neck of the flask. The supports for this asbestos sheet may conveniently consist of triangular pieces of metal sheet firmly fixed to the screen at its four corners.

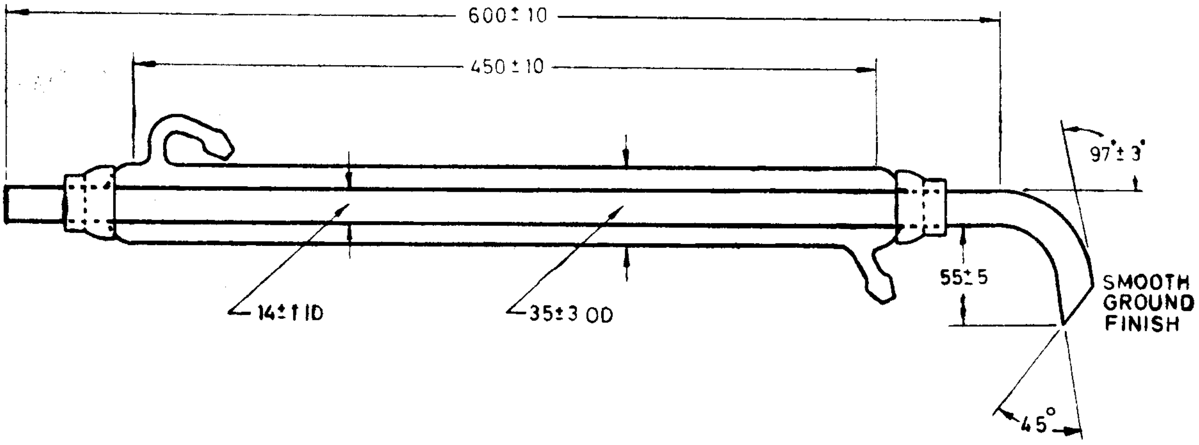
c) In one of the narrow sides of the screen, a door shall be provided having the dimensions and position as shown in Fig. 3. In each of the narrow sides of the screen, a window shall be placed centrally, with a bottom of the window on a level with the top of the asbestos sheet. The dimensions and position of the windows are shown in Fig. 3.

d) In addition to the asbestos sheet referred to above, an asbestos board, 150 mm square, is also required. This additional asbestos board shall be 8 mm in thickness and shall have a central hole 50 mm in diameter. This shall be placed on the asbestos sheet described above.

A-5.1.6 *Electric Heater or Gas Burner or Other Flame Type of Heater* — Any type of heater or burner that enables the distillation to be carried out as described in **A-5.2** may be employed.

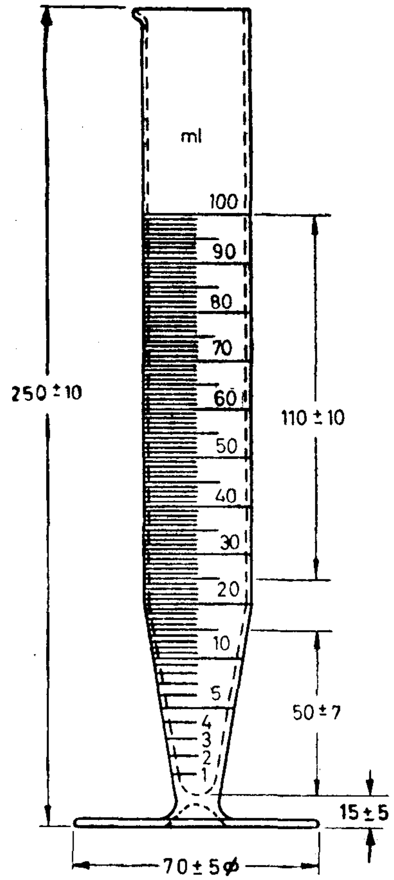
A-5.2 *Procedure*

Assemble the apparatus as shown in Fig. 5. Measure 100 ml of the material at laboratory temperature in the receiver and transfer it to the distillation flask, the contents of the receiver being allowed to drain for 15 seconds into the



All dimensions in millimetres.

Fig. 1 Liebig Condenser



All dimensions in millimetres.

FIG. 2 RECEIVER

flask. Add a fragment of pumice stone or other suitable inert material to prevent bumping. Place the flask, thermometer and receiver in position. Apply heat at a uniform rate so regulated that the first drop of distillate falls from the end of the condenser in 12 or 13 minutes. Further regulate the heat so that the distillate is collected at the rate of 3 to 4 ml per minute. Record the temperature readings when 2 percent and 98 percent of the volume taken has been distilled over and collected in the receiver.

NOTE — It is desirable to run a preliminary distillation so that the heat source may be regulated to supply heat to distil the liquid at the specified rate. The flask should be cleaned after preliminary distillation.

A-5.2.1 *Correction of Thermometer Reading* — The following corrections shall be applied.

A-5.2.1.1 *Error of scale* — In all thermometer readings, make the correction as indicated on the certificate of the instrument.

A-5.2.1.2 *Correction for barometric pressure* — If the barometric pressure prevailed during the determination is 760 mm Hg, no correction need be applied to the specified temperature readings, and the temperature scale as corrected under **A-5.2.1.1** shall be used as such. If, however, the prevailing barometric pressure deviates from 760 mm Hg, the specified temperature shall be corrected as follows:

a) For every 10 mm above 760 mm Hg, substract 0·15°C from the specified temperature;

b) For every 10 mm below 760 mm Hg, add 0·15°C to the specified temperature.

NOTE — These corrections are valid only for pressure above 700 mm Hg.

A-5.3 Report

Report whether the difference between the temperature (running points) at which 2 and 98 percent of the volume taken have been collected (*see* **A-5.2** and **A-5.2.1**) and the range, meets the requirement [*see* Table 1 Sl No. (iv)].

A-6 DETERMINATION OF ASH

A-6.0 General

The material is heated at 500°C to 600°C and ash determined.

A-6.1 Procedure

Evaporate about 100 ml of the material into a tared platinum or silica dish. Heat gently at first, evaporate to dryness and then ignite to almost ash. Then heat in an electric furnace at 500°C to 600°C for about 30 minutes, allow to cool in a desiccator and weigh. Repeat the heating and cooling till the difference between two successive weighings fall within 0·5 mg.

A-6.2 Calculation

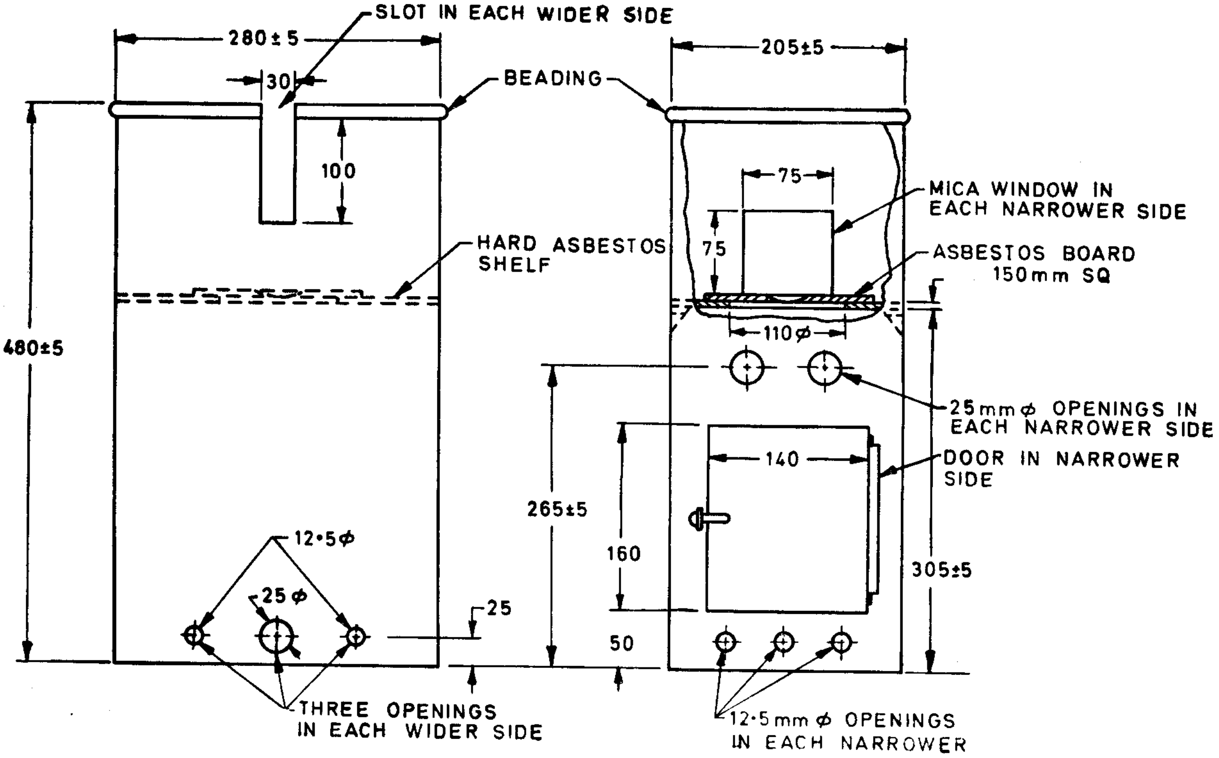
$$\text{Ash, percent by mass} = \frac{M}{V \times \rho} \times 100$$

where

M = mass in g of the ignited residue;

V = volume of the material taken for the test; and

ρ = density in g/ml, of the material taken for the test.



All dimensions in millimetres.

FIG. 3 RECTANGULAR DRAUGHT SCREEN

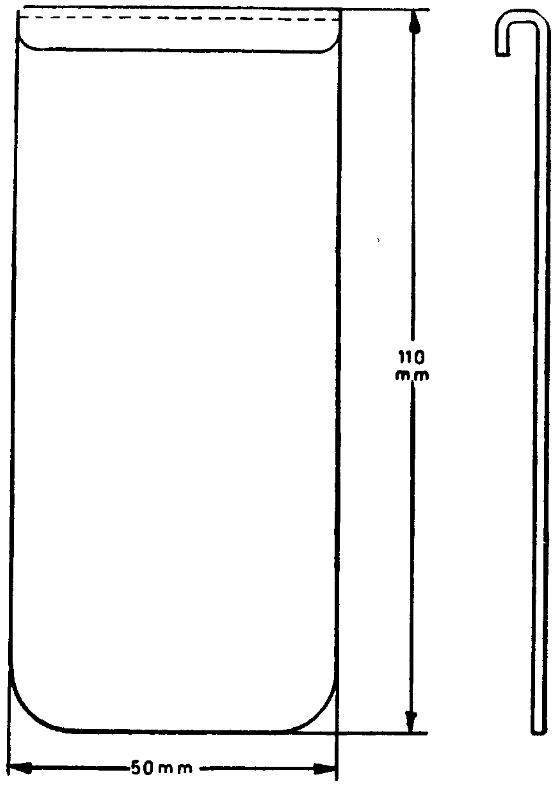


FIG. 4 REMOVABLE SHUTTER

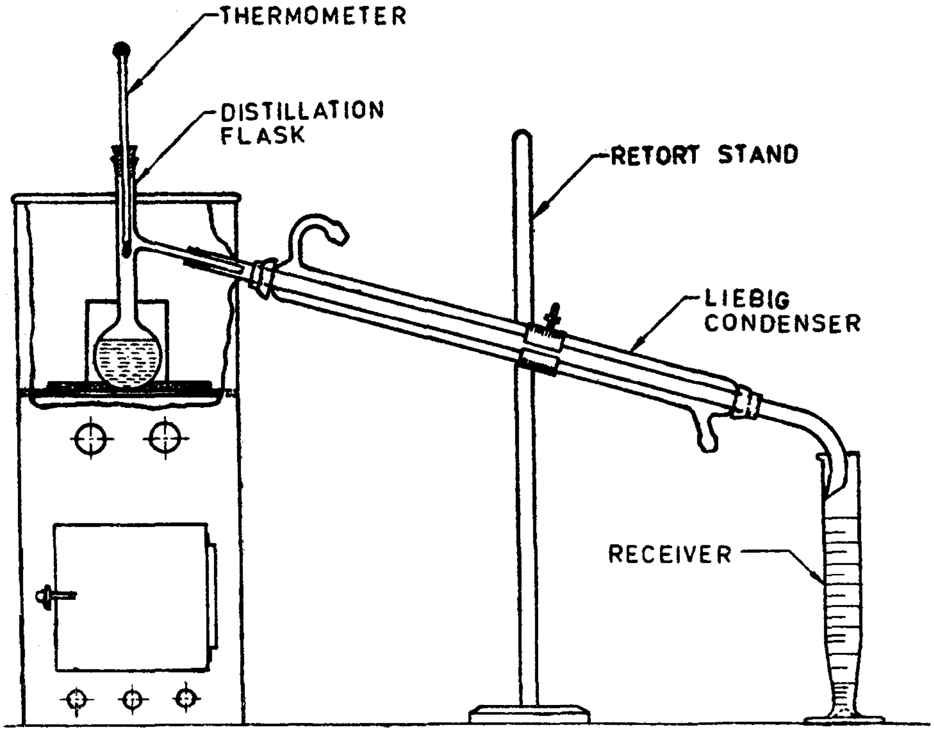


FIG. 5 ASSEMBLY OF APPARATUS

ANNEX B

(*Clause* 6)

SAMPLING

B-1 GENERAL REQUIREMENTS

B-1.0 While handling and testing the samples, in addition to the precautions in handling given in **4** of the specification, following precautions shall be observed.

B-1.1 Samples shall be taken in a protected place not exposed to damp air, dust or soot.

B-1.2 The sampling instrument shall be clean and dry.

B-1.3 Precuations shall be taken to protect the samples, the material being sampled, the sampling instrument and containers for samples from adventitious contamination.

B-1.4 To draw a representative sample, the contents of each drum selected for sampling shall be mixed as thoroughly as possible by suitable means.

B-1.5 The samples shall be placed in suitable, clean, dry and air-tight glass bottles or other containers on which the material has no action.

B-1.6 The sample containers shall be of such a size that they are almost completely filled by the sample.

B-1.7 Each sample container shall be sealed air-tight after filling and marked with full details of sampling, the date of sampling and other details given as under **5.2** (c) to (e).

B-2 SCALE OF SAMPLING

B-2.1 Lot

All the drums in a single consignment of the material drawn from the same batch of manufacture shall constitute a lot.

B-2.2 For ascertaining the conformity of the material in a lot to the requirements of the specification, samples shall be tested from each lot separately.

B-2.3 The number of drums to be selected from the lot for this purpose, shall depend on the size of the lot and shall be in accordance with Table 2.

B-2.3.1 These drums shall be selected at random from the lot. In order to ensure the randomness of selection, procedures given in IS 4905 : 1968 may be followed.

B-3 TEST SAMPLES AND REFEREE SAMPLES

B-3.1 From each of the drums selected according to **B-2.3**, a representative portion of the material from different parts of the drums shall be drawn. Each of these individual portions shall be mixed thoroughly so as to give a representative sample for the drum.

B-3.2 From the samples (*see* **B-3.1**), representing different drums, a small but approximately equal quantity of material shall be taken and thoroughly mixed to constitute a composite sample of not less than 1 200 ml in volume. The composite sample shall be divided into three equal parts, one for the purchaser another for the supplier and the third to be used as a referee sample. These shall be transferred to separate containers which shall then be sealed air-tight and labelled with full identification particulars given in **B-1.7**.

B-3.3 A referee sample shall bear the seals of both the purchaser and the supplier and shall be kept at a place agreed to between the two. This shall be used in case of any dispute between the two.

B-4 NUMBER OF TESTS

Tests for determination of all the requirements given in **3.1** and Table 1 shall be carried out on the composite sample.

B-5 CRITERIA FOR CONFORMITY

The lot shall be declared as conforming to the requirements of the specification if all the test results on the composite sample meet the corresponding requirements given in **3.1** and Table 1.

Table 2 Scale of Sampling

(*Clause* B-2.3)

Lot Size	Number of Drums to be Selected
(1)	(2)
Up to 25	3
26 to 50	4
51 to 100	5
101 to 300	7
301 and above	10

Standard Mark

The use of the Standard Mark is governed by the provisions of the *Bureau of Indian Standards Act, 1986* and the Rules and Regulations made thereunder. The Standard Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well defined system of inspection, testing and quality control which is devised and supervised by BIS and operated by the producer. Standard marked products are also continuously checked by BIS for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

Bureau of Indian Standard

BIS is a statutory institution established under the *Bureau of Indian Standards Act, 1986* to promote harmonious development of the activities of standardization, marking and quality certification of goods and attending to connected matters in the country.

Copyright

BIS has the copyright of all its publications. No part of these publications may be reproduced in any form without the prior permission in writing of BIS. This does not preclude the free use, in the course of implementing the standard, of necessary details, such as symbols and sizes, type or grade designations. Enquiries relating to copyright be addressed to the Director (Publications), BIS.

Revision of Indian Standards

Indian Standards are reviewed periodically and revised, when necessary and amendments, if any, are issued from time to time. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition. Comments on this Indian Standard may be sent to BIS giving the following reference:

Doc : No. PCD 9 (60)

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

BUREAU OF INDIAN STANDARDS

Headquarters :

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110002
Telephones : 331 01 31, 331 13 75
Telegrams : Manaksanstha
(Common to all Offices)

Regional Offices :	Telephone
Central : Manak Bhavan, 9 Bahadur Shah Zafar Marg NEW DELHI 110002	311 01 31 331 13 75
Eastern : 1/14 C. I. T. Scheme VII M, V. I. P. Road, Maniktola CALCUTTA 700054	37 86 62
Northern : SCO 445-446, Sector 35-C, CHANDIGARH 160036	53 38 43
Southern : C. I. T. Campus, IV Cross Road, MADRAS 600113	235 02 16
Western : Manakalaya, E9 MIDC, Marol, Andheri (East) BOMBAY 400093	6 32 92 95

Branches : AHMADABAD, BANGALORE, BHOPAL, BHUBANESHWAR, COIMBATORE, FARIDABAD, GHAZIABAD, GUWAHATI, HYDERABAD, JAIPUR, KANPUR, LUCKNOW, PATNA, THIRUVANANTHAPURAM.

Printed at Printwell Printers, Aligarh, India